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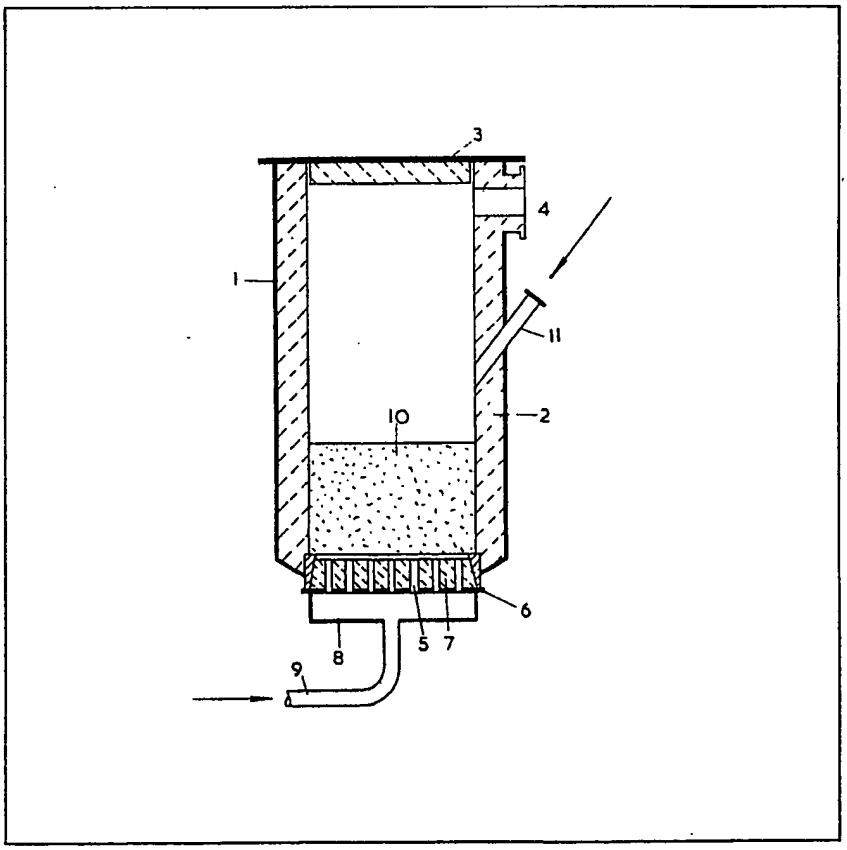
# (54) Refractory lined vessel and method of use

(57) A chlorinator for use in the chlorination of a titaniferous material in which a bed of the material is maintained in fluid suspension during the chlorination reaction and which is retained on a base having individual means to introduce and distribute chlorine gas. The vessel is provided with a lining at least a part of which is formed of a refractory concrete which can also be used to form the individual means to introduce the chlorination gas and a refractory material between these means.

The refractory concrete is one containing an aggregate having a particle size of less than 12 mm and formed of a calcined clay containing aluminium in an amount when expressed as $Al_2O_3$ of from 43% to 63% by weight and silicon when expressed as $SiO_2$ of from 53% to 33% by weight of the aggregate. The refractory concrete also contains finely-dividied corundum and an aluminium phosphate binding agent.

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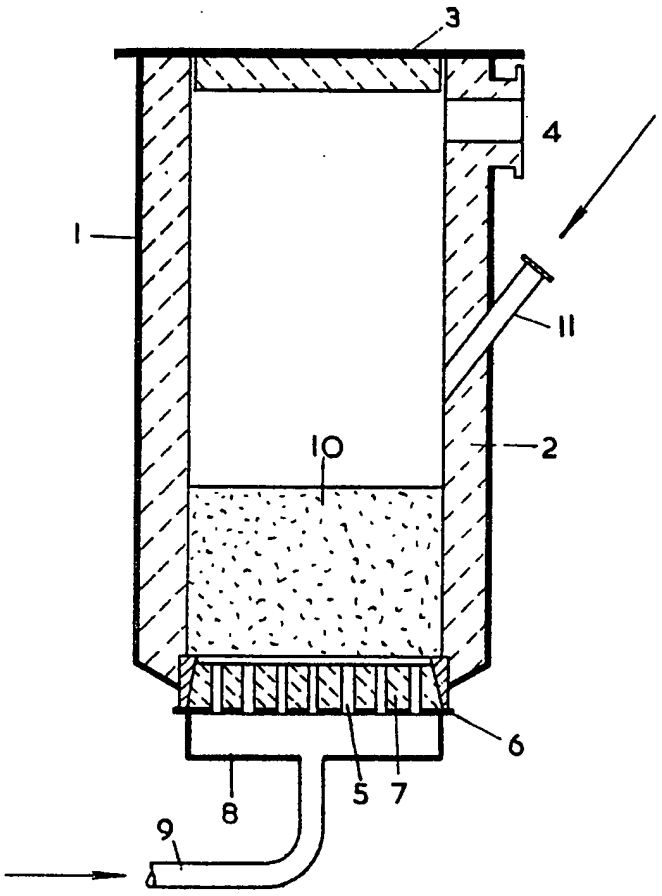
The drawing(s) originally filed was/were informal and the print here reproduced is taken from a later filed formal copy.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SPECIFICATION

**Refractory lined vessel and method of use**

This invention relates to a refractory lined vessel, particularly a chlorinator, and its method of use.

During use of a chlorinator for chlorination of a titaniferous material existing refractory materials exhibit wear when used as linings and this necessitates expensive repair or replacement at frequent intervals. There is a need for such chlorinators having longer lives than at present between repairs.

According to the present invention a vessel suitable for use in the chlorination of a titaniferous material comprises an outershell-like retaining wall having a closed upper end and a base, inlet means to introduce a titaniferous material into the vessel and outlet means located at or adjacent the upper end for the discharge of gaseous reaction products, said base having means to introduce a gas into the vessel and to distribute said gas over the horizontal cross-sectional area of the vessel, said retaining wall having a refractory lining and said base having refractory infill material between individual means to introduce and to distribute said gas and, said individual means, said lining and/or said infill material being formed of a refractory concrete containing an aggregate having a particle size of less than 12 mm in diameter and being formed of a calcined clay containing aluminium in an amount when expressed as $Al_2O_3$ of from 43% to 63% by weight and silicon when expressed as $SiO_2$ of from 53% to 33% by weight of the aggregate and other minor components forming the balance of the aggregrate, finely-divided corundum in an amount of at least 0.5% of the weight of the concrete and having a particle size less than 50 microns and an aluminium phosphate binding agent in an amount of from 2% to 10% by weight of the concrete when expressed as aluminium orthophosphate.

Preferably the binding agent is present in an amount of from 4% to 6% by weight of the concrete as aluminium orthophosphate.

The aggregate preferably is a calcined flint clay having a particle size of not greater than 10 mm in diameter and most preferably a particle size not greater than 8 mm in diameter. The most preferred form of aggregate contains aluminium as $Al_2O_3$ in an amount of from 51% to 57% by weight and silicon in an amount of from 47% to 41% by weight as $SiO_2$ both based on the weight of aggregate.

The refractory concrete also contains corundum as stated in an amount of at least 0.5% by weight of the concrete. Usually the amount of corundum will not exceed 10% by weight and preferably the amount of corundum is from 1% to 5% by weight of the concrete. The particle size of the corundum is less than 50 microns and preferably less than 45 microns.

The concrete mixture is prepared by mixing in the chosen proportions the desired ingredients together with the addition of sufficient water to allow the mixture to be worked and applied to the surfaces and to permit setting to take place. The mixture can be applied by casting, gunning, trowelling or by extrusion as is appropriate.

The vessel is of particular use in the chlorination of a titaniferous material to produce titanium tetrachloride. The chlorination is carried out in the presence of a carbonaceous reducing agent, e.g. coke, by chlorine gas passing upwardly through a bed of the reactants at such a rate and density as to maintain the bed in fluid suspension. Normally the bed of material is heated to at least 800°C to initiate the chlorination reaction.

The bed of titaniferous material, which most suitably is ilmenite or more preferably mineral rutile, is retained within the chlorinator on the base formed by an array of gas distribution tubes or tuyeres carried by a lower support plate. Refractory infilling material is provided between the gas distributions tubes. The gas distribution tubes or tuyeres can be formed of a refractory material similar to that used to form the lining. Normally a wind box or gas distribution chest is located below the array of gas distribution tubes so that as to fluidise the bed and to effect the chlorination reaction can be fed to the tubes from a suitably located inlet.

One form of chlorinator constructed in accordance with the invention will now be described by way of example only with reference to the attached drawing which is a diagrammatic section of a suitable vessel.

The chlorinator consists of an outer shell 1 having a refractory lining 2. The upper end of the shell 1 is closed by a lined lid 3 and the shell 1 has an upper outlet 4 for chlorination products. Across the base of the shell 1 is positioned an array or an assembly of gas distribution tubes 5 carried on a support plate 6 with the spaces between adjacent tubes 5 being filled by refractory infill 7 similar to that forming the lining 2. Below the support plate 6 is a wind box or gas distribution chest 8 having an inlet 9 for a gas.

In use the bed 10 of the titaniferous material to be chlorinated is carried by the array of tubes 5 and an inlet port 11 is provided through which the bed may be replenished in use.

The invention also includes a method of chlorination of a titaniferous material to produce titanium tetrachloride which comprises establishing a bed of a titaniferous material to be chlorinated and a reducing agent on the base of a vessel in accordance with the invention and heating the bed up to the desired reaction temperature and passing chlorine gas through the gas distribution tubes to react with the titaniferous material and to maintain the bed in fluid suspension and collecting the titanium tetrachloride produced by the chlorination.

A vessel having a lining formed of a refractory concrete in accordance with the invention is expected to have a life of at least 10 times as long as one with a lining formed of a high alumina cement bound aggregate.

CLAIMS (Filed on 9/3/83)

1. A vessel suitable for use in the chlorination of a titaniferous material which comprises an outer shell-like retaining wall having a closed upper end and a base, inlet means to introduce a titaniferous material into the vessel and outlet means located at or adjacent the upper end for the discharge of

gaseous reaction products, said base having means to introduce a gas into the vessel and to distribute said gas over the horizontal cross-sectional area of the vessel, said retaining wall having a refractory lining and said base having refractory infill material between individual means to introduce and to distribute said gas and, said individual means, said lining and/or said infill material being formed of a refractory concrete containing an aggregate having a particle size of less than 12 mm in diameter and being formed of calcined clay containing aluminium in an amount when expressed as $Al_2O_3$ of from 43% to 63% by weight and silicon when expressed as $SiO_2$ of from 53% to 33% by weight of the aggregate and other minor components forming the balance of the aggregate, finely-divided corundum in an amount of at least 0.5% of the weight of the concrete and having a particle size less than 50 microns and an aluminium phosphate binding agent in an amount of from 2% to 10% by weight of the concrete when expressed as aluminium orthophosphate.

2. A vessel according to claim 1 in which the said binding agent is present in an amount of 4% to 6% when expressed as aluminium orthophosphate by weight of the concrete.

3. A vessel according to claim 1 or 2 in which the aggregate is a calcined flint clay having a particle size of not greater than 10 mm.

4. A vessel according to claim 3 in which the aggregate has a particle size of not greater than 8 mm in diameter.

5. A vessel according to any one of claims 1 to 4 in which the aggregate contains aluminium as $Al_2O_3$ in an amount of from 51% to 57% and silicon in an amount of from 47% to 41% by weight as $SiO_2$ on weight of aggregate.

6. A vessel according to any one of the preceding claims in which the amount of said corundum does not exceed 10% by weight of said concrete.

7. A vessel according to claim 6 in which the amount of said corundum is from 1% to 5% by weight of said concrete.

8. A vessel according to any one of the preceding claims in which the corundum has a particle size less than 45 microns.

9. A method of chlorination of a titaniferous material to produce titanium tetrachloride which comprises establishing a bed of titaniferous material mixed with a carbonaceous reducing agent on the base of a vessel according to claim 1, heating the bed to at least 800°C and passing chlorine gas upwardly through said bed to maintain the bed in fluid suspension and produce the desired titanium tetrachloride.

11. A vessel constructed and arranged substantially as described herein and shown in the accompanying drawing.

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